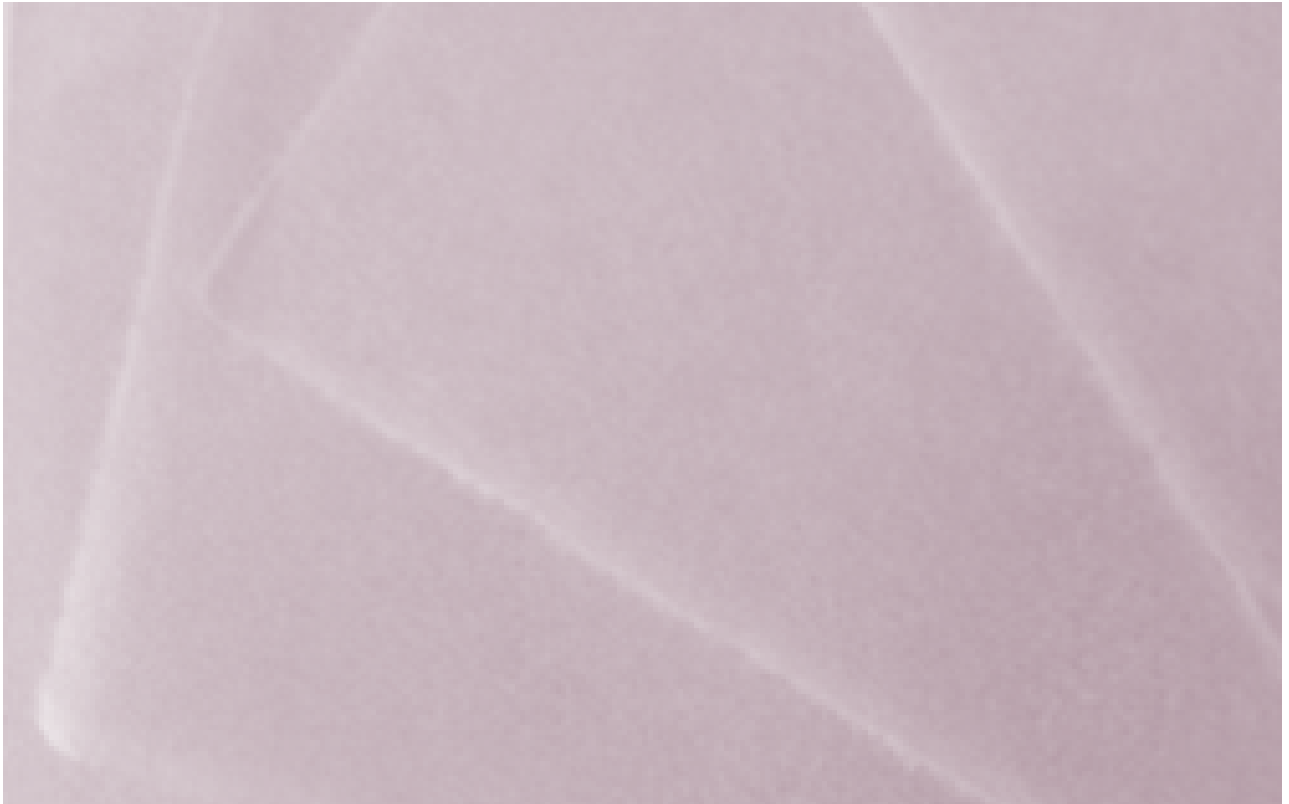mistra
future
fashion

paper-based nonwoven textiles - material recycling and industrial composting

by

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1. introduction

Within the Mistra Future Fashion program, the possibility to produce textile-like materials on a paper machine was explored. Here the overall goal is to find a wearable material that leaves a small footprint throughout the value cycle[1]. The idea was to have a material with a fast production line (compared to weaving or knitting), limited washing and handling during the user phase, and easy end of life processing. The scope of project can be visualized as part of the Ellen MacArthur Foundation circular economy model (figure 1).

The consumers' experience of the textile-like material is important, that is why it is natural to consider the materials conformity to properties that are usually associated with textiles, such as softness, handfeel, flexibility, stretch, strength, drape and a sound dampening effect. To be able to influence the consumers perception and final acceptance of the material, there should be finishing and conversion processes that supports a large span of possible visual and haptic expressions.

From a sustainability perspective, it is important to limit the resources used to manufacture the base material, the processing into a final garment, the footprint during use phase and to consider viable end-of-life routes[1].
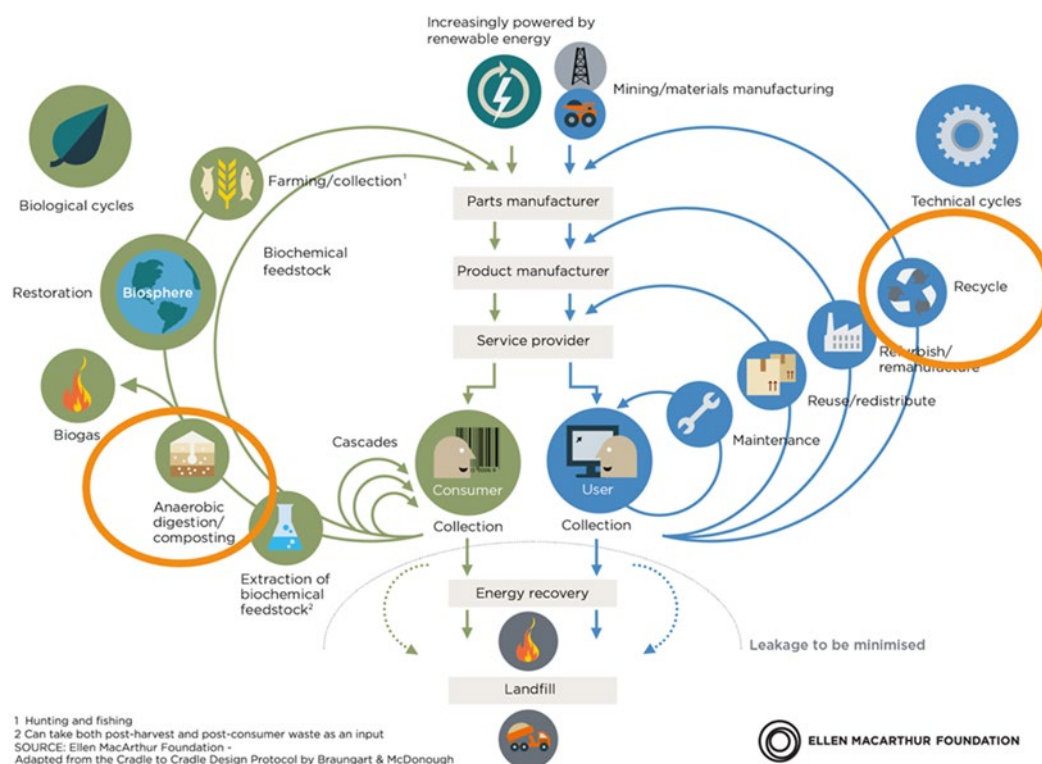
figure 1 Illustration of the scope of project work using Ellen MacArthur Foundation circular economy model. Industrial composting of the fast fashion material as a part of the biological cycle and material recycling of the fast fashion material as a part of the technical cycle.[2]

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[1] Granberg H., Zachrisson A-M., Granlöf L., Mistra Future Fashion report Crimping and welding of textile-like papers, RISE Bioeconomy, 2018.
[2] Ellen MacArthur Foundation, Rethink the future 1, Towards the circular economy, Economic and business rationale for an accelerated transition, 2013.

In the current study we have chosen to work with a mixture of bio-based materials as stock to the paper machine[1]:

- Polylactide staple fiber (polylactic acid, PLA fiber), is soft and has thermoplastic properties that can be exploited in a thermic welding finishing process to increase material strength.
- Sulphate softwood paper pulp is used because of its low footprint compared to staple fibers, and
- Microfibrillated cellulose from wood in small quantities is used to increase the material strength.

The mixing of the components followed two distinctly different routes, starting with:

- Material A with relatively high PLA fiber content (57%) resulting in a soft, stretchy but weak material, in two variations: base material A and material A with welding finishing.
- Material B with low PLA fiber content (5%) resulting in a stiff but strong material, in two variations: base material B and material B with welding finishing.

It is envisioned that the route A-materials (high PLA content) could be industrially composted and that the route B-materials (low PLA content) could be materially recycled as a paper or paper board[1] (see Figure 1).

The task 1.1.5 ´Recycling models for Short-Life Garments´ focused on material recycling of the innovative material B, though material A was also tested. Thus, both materials, A and B, were re-slushed into its fiber components in order to understand possibilities and limitations of the material as a function of PLA content and of welding finishing process.

The task SRF.1.2 'Industrial composting of paper-based non-woven textiles' focused on the biodegradation of the innovative material A in two variations: base material A and material A with welding finishing.

The results of both tasks 1.1.5 and SRF.1.2, are compiled into the current report.

1.1. objectives

The objective of this study is to, in a practical way:

- Explore key factors in production of paper-based nonwoven textiles that support their recyclability as paper or board.
- Evaluate full industrial compostability, including biodegradability, of the paper-based nonwoven textiles, defining whether this is a possible end-of-life scenario.

The expected new knowledge is translation of design and material insights into material samples. This will add an extra layer of knowledge to designing of garment prototypes, i.e. through material understanding.

2. material

All material samples analysed had same grammage of 90 g/m^2, while containing varied proportions of PLA (polylactic acid), unbleached sulphate pulp, and CNF (cellulose nanofibrils), see table 1.

Welding (W) finishing of the materials A-W and B-W was performed using a hot tip in a 3D-printer Ultimaker. In particular, square welding pattern was applied, having a maximum of 2 mm between each welding spot (3 mm center-to-center-distance)[1].

table 1 The material samples A, A-W as well as B and B-W were analyzed in the material recycling study. The material samples A and A-W were the industrial composting study.

Sample #	Aimed material grammage (g/m2)	Material composition	In recycling study	In industrial composting study
A	90	40% unbleached sulphate pulp (unrefined), 57% PLA, 3% CNF – base material	V	V
A-W	90	40% unbleached sulphate pulp (unrefined), 57% PLA, 3% CNF – base material with welding finishing	V	V
B	90	95% unbleached sulphate pulp (refined), 5% PLA, 0% CNF – base material	V	-
B-W	90	95% unbleached sulphate pulp (refined), 5% PLA, 0% CNF – base material with welding finishing	V	-

*A, B – base materials, A-W, B-W – base material with welding finishing

In the beginning of the project, screening recycling studies were performed on several base material A samples with different grammage but same material composition. This was done in order to understand whether base material A grammage will affect recycling ability of the material. In the same manner, in the screening accelerated ageing study, the base material A grammage was also varied, while the material composition remained the same, see table 2 and table 3, respectively.

table 2 Screening study material prior to the recycling study. A – base material.

Sample #	Grammage (g/m2)	Material sample composition
A-1	42,4	40% unbleached sulphate pulp, 57% PLA, 3% CNF – *base material*
A-6	229	40% unbleached sulphate pulp, 57% PLA, 3% CNF – *base material*

table 3 Screening material for accelerated ageing study prior to the recycling study. A – base material.

Sample #	Grammage (g/m2)	Material sample composition
A-1	42,4	40% unbleached sulphate pulp, 57% PLA, 3% CNF
A-2	76,6	40% unbleached sulphate pulp, 57% PLA, 3% CNF
A-3	162,1	40% unbleached sulphate pulp, 57% PLA, 3% CNF
A-4	211,1	40% unbleached sulphate pulp, 57% PLA, 3% CNF
A-6	229	40% unbleached sulphate pulp, 57% PLA, 3% CNF
A-8	164,8	40% unbleached sulphate pulp, 57% PLA, 3% CNF

2.1. material recycling

The material recycling study focuses on re-slushability of the material A and B samples produced at the semi-pilot paper machine STRATEX, in accordance with ISO 5263[3] and Tappi T275 sp – 98[4].

Definition of re-slushability of paper: The percentage (%) of weighed amount of paper that after wet disintegration and subsequent screening does not stick on screen plates with slit widths 0.15 mm[3]:

$$U = \frac{a - v}{a} \times 100$$

whereas:
U = Re-slushability, %
v = Dry weight of screening residue, g
a = Dry weight of paper, g

In the screening material recycling study, where the base material A grammage was varied and the material composition was constant, the fibers of the re-slushed material samples were captured back / collected for the second production cycle with aim to produce lab sheets of size 15 cm x 15 cm from the re-slushed / recovered fibers.

table 4 Preparation of recycled material samples (2nd production cycle, at lab)

Sample # Units	Grammage g/m2	Concentration g/l	Sheet weight g	Fiber suspension g
A-1	44.0	3.144	1.166	371
A-6	240.9	3.152	6.298	1998

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[3] ISO 5263:2004 Pulps - Laboratory wet disintegration, 2004.
[4] T 275 sp-98 Screening of pulp (Somerville-type equipment), 1998

2.2. accelerated ageing

The screening accelerated ageing study was performed in the climate chamber at the conditions below in order to simulate the temperature and humidity of the industrial composting (though in the air environment):

Temperature: 58°C
Relative humidity: 65% RH
Ageing time: 0h, 500h and 1000h

2.3. industrial composting

To determine if a material is compostable according to EN 13432[5], the four required assessment criteria below need to be fulfilled:

- Elementary analysis (chemical composition): The standard sets limits for volatile matters, heavy metals and fluorine. Amounts allowed per weight unit must not be exceeded. (Chemical analysis according to several ISO standards).

- Biodegradation: Chemical breakdown of material into carbon dioxide, water and minerals. Pursuant to the standard at least 90% of the material has to be broken down by biological action within 6 months (ISO 14855).

- Disintegration: The physical decomposition of a material into tiny pieces. After 12 weeks at least 90% of the material should be able to pass through a 2 x 2 mm mesh (ISO 20200 lab scale, ISO 16929 pilot scale).

- Quality of the final compost and eco-toxity: the quality of the compost should not decline as a result of the added material. The standard specifies checking this via toxicity tests involving making an examination to see if the germination and biomass production of plants are not adversely affected by the influence of composted material (OECD guideline 208 based internal RISE method).

The industrial composting test performed in the project included the first two assessment criteria stated above (chemical composition and biodegradation) due to high interest in testing of industrial composting dynamics of base material and material with welding finishing, i.e. material A and A-W. Thus, it was decided to test the visual analysis of the compost instead of performing full disintegration analysis, and quality of the final compost and eco-toxicity. According to the standard[5], if both criteria are met, i.e. chemical composition and biodegradation, a material can be classified as biodegradable.

Experimental Part 1 – Elementary analysis (chemical composition)

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[5] European standard EN 13432 Packaging – requirements for packaging recoverable through composting and biodegradation – Test scheme and evaluation criteria for the final acceptance of packaging, 2000.

The industrial composting result are required to be reported in terms of elements analyzed and respective threshold values according to EN 13432 as shown in table 5.

table 5 Threshold levels in the elementary analysis

Element	mg/kg	Element	mg/kg
Zn	<4	**Hg**	<0,2
Cu	<2	**Cr**	<1
Ni	<1	**Mo**	<0,4
Cd	<0,2	**Se**	<0,6
Pb	<0,2	**As**	<4
		F	<100

Experimental Part 2 – Biodegradation
The tested materials A and A-W were cut into small pieces. A mixture of mature compost and structural material (vermiculite) was prepared and the materials to be tested and the reference material (benchmark) were added. All test vessels were installed with oxygen and no CO_2 availability, at high temperature (58 °C).

The amount of carbon dioxide developed was measured during 6 months, with the starting date 2018-05-18. The developed amount of carbon dioxide was then compared with the theoretical maximum, based on the organic content of the material, and a percentage biodegradability was obtained.

3. results and discussion

In this section, the results of the material recycling and industrial composting are presented. In addition, the results from the screening material recycling and accelerated ageing test are also reported.

3.1. material recycling

The re-slushability procedure is visualized in figure 2, including steps of material sample bloating, reslushing and screening.
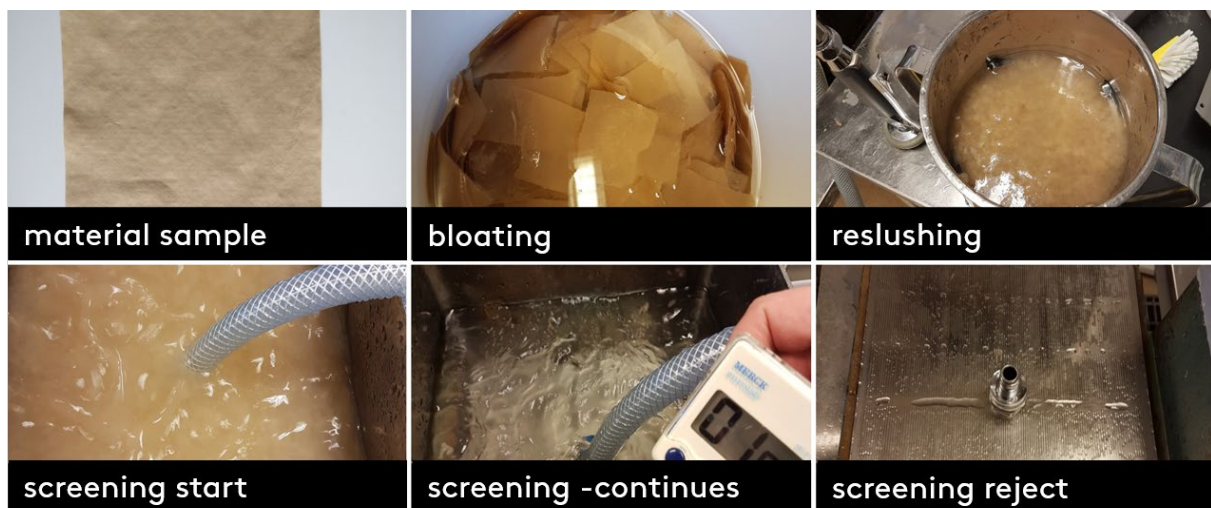

figure 2 Material recycling (reslushability) test visualization.

The results of the material recycling study in terms of re-slushability are shown in table 6. The re-slushability results showed that the base materials A and B are 99,9 % reslushable, i.e. can be disintegrated back into fiber components.

Base materials A and B treated with welding finishing however showed different results. While material A with welding finishing (see figure 5) was reslushable to only 55,3%, the material B with welding finishing (see figure 6) was almost completely reslushable, 99,64%. figure 7 and figure 8 show rejects from the treated material A and material B, respectively.

table 6 Re-slushability test results. A, B – base materials, A-W, B-W – base material with welding finishing.

Sample #	Aimed material grammage (g/m2)	Material composition	Reslushability
A	90	40% unbleached sulphate pulp (unrefined), 57% PLA, 3% CNF – base material	99,9
A-W	90	40% unbleached sulphate pulp (unrefined), 57% PLA, 3% CNF – base material with welding finishing	55,30
B	90	95% unbleached sulphate pulp (refined), 5% PLA, 0% CNF – base material	99,9
B-W	90	95% unbleached sulphate pulp (refined), 5% PLA, 0% CNF – base material with welding finishing	99,64

The material A-W reject was analyzed and contained 61% PLA, the rest of the reject material were pulp fibers that were melted into PLA during welding finishing.

Figure 3 Material A - base material.

figure 4 Material B - base material.

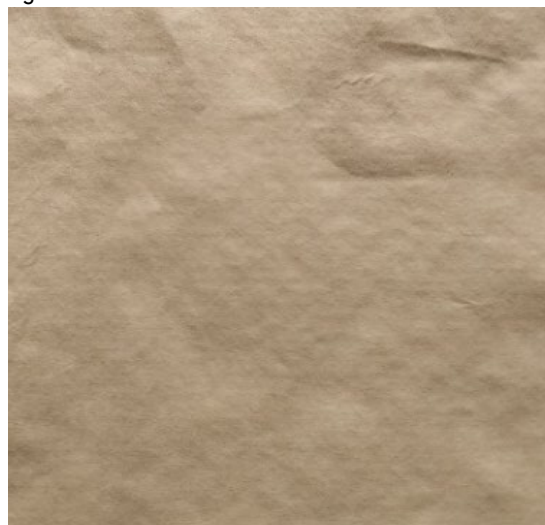figure 5 Material A – base material with welded finishing.

figure 6 Material B – base material with welded finishing.

figure 7 Reject from re-slushing of material A with welded finishing.

figure 8 Reject from re-slushing of material B with welding finishing.

The screening material recycling test of the base materials showed similar trend.

table 7 Re-slushability test results from the screening material recycling test.

Sample #	Grammage (g/m2)	Material sample composition	Reslushability
A-1	42,4	40% unbleached sulphate pulp, 57% PLA, 3% CNF – *base material*	99,85
A-6	229	40% unbleached sulphate pulp, 57% PLA, 3% CNF – *base material*	99,92

The re-slushability results showed that both base material A samples were over 99% re-slushable, meaning that the material grammage did not affect material recyclability.

In the next step, after the material samples were re-slushed back into fibers, these fibers were recycled into new material samples. Thus, a second production cycle took place. It is important to notice that the first production cycle was done at the pilot-scale machine STRATEX where the rolls of material samples were produced, while the second production cycle was done at the lab-scale where lab sheets were produced, due to the low amount of fibers available for experiments. As can be seen in figure 9 a) and b), the lab sheets have more homogeneous structure.

a)   b)

figure 9: lab sheets of a) Above: Sample 1 – First production cycle at STRATEX pilot-scale machine. Below: Sample 1 - Second production cycle as material at lab (lab sheet), and b) Above: Sample 6 – First production cycle at STRATEX pilot-scale machine., Below: Sample 6 - Second production cycle as material lab (lab sheet).

3.2. accelerated ageing

The preliminary accelerated ageing study included visual analysis of the material samples A-1 – A-6 and analysis of their respective mechanical properties.

The analysis has not shown any visible signs of material ageing at the studied time periods of 500h and 1000h, neither in material machine direction (MD) nor cross direction (CD), see figure 10 and Appendix.

Ageing: 0h – machine direction

Ageing: 0h – cross direction

Ageing: 500h – machine direction

Ageing: 500h – cross direction

Ageing: 1000h – machine direction

Ageing: 1000h – cross direction

figure 10: Visual analysis of the aged material sample A-1 (grammage 42,4 g/m²). Results from the screening accelerated ageing study.

No significant change in mechanical properties of the material samples A-1 – A-6 were observed after 1000 hours of accelerated ageing (see figure 11 - figure 13). Likely, some degree of degradation in the PLA occurred but not enough to impact the mechanical properties of the materials.
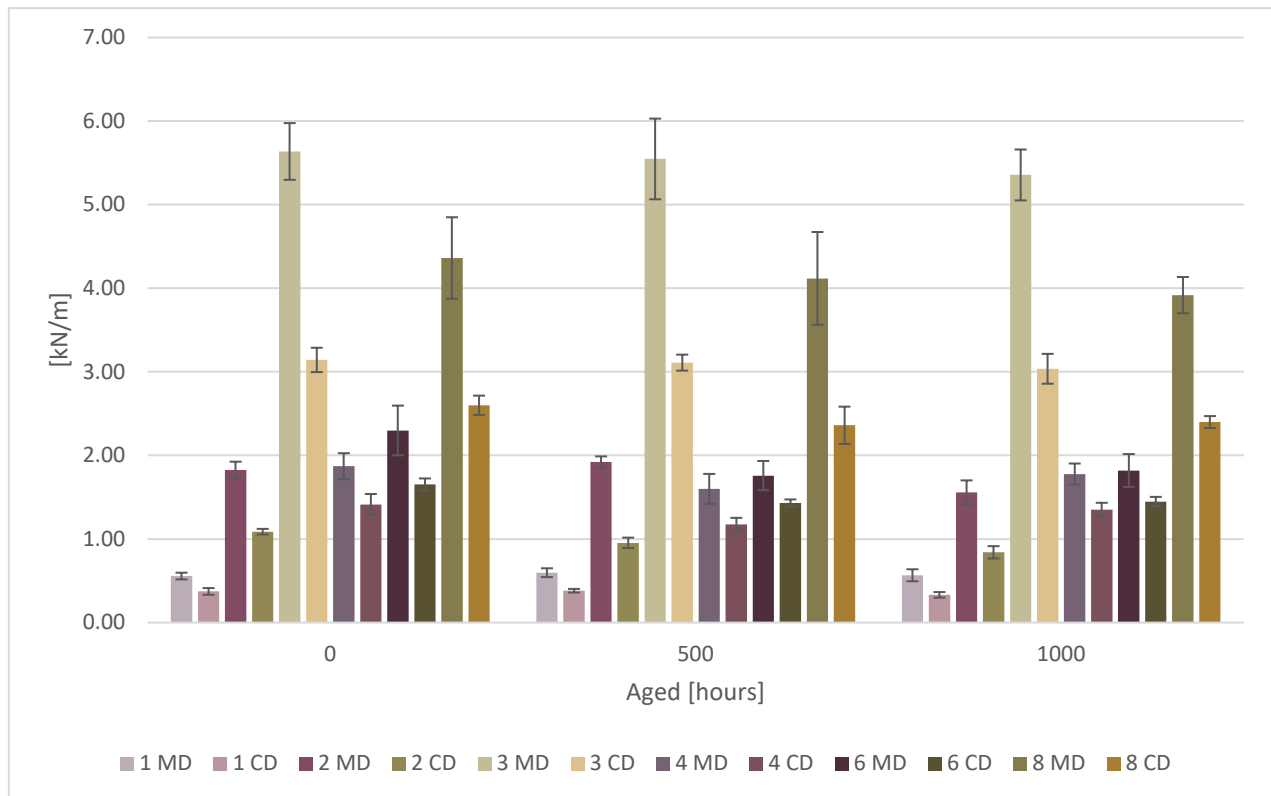
figure 11 Tensile strength of the material samples A-1 – A-8 with varied grammage.
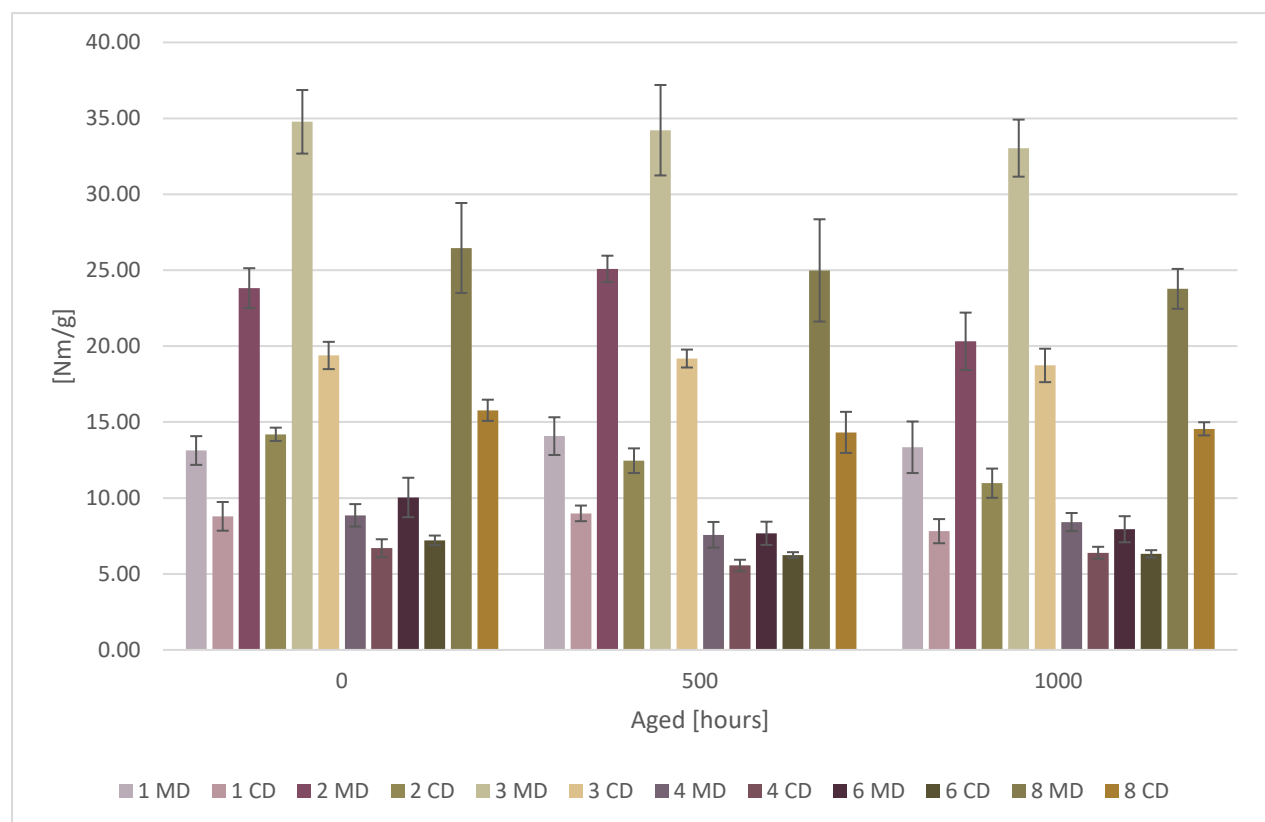
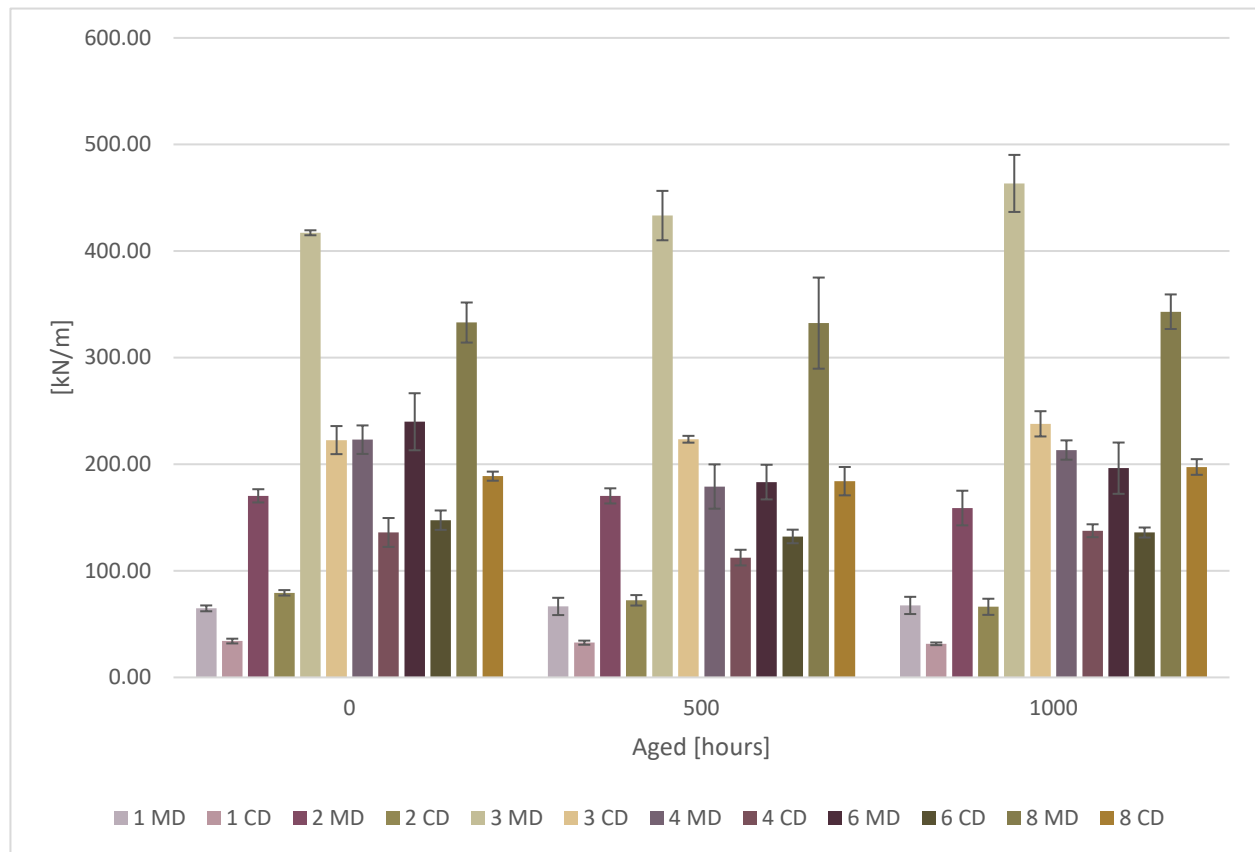figure 12 Tensile index of the studied material samples A-1 – A-8 with varied grammage.

figure 13 Tensile stiffness of the studied material samples A-1 – A-8 with varied grammage.

3.3. industrial composting

Part 1 – Elementary analysis (chemical composition)

Analysis of ash in material samples A and A-W (550 °C, weight-%) showed that only 0.2 % of the tested materials consisted of inorganic matter. Hence, further analysis of metals was not necessary because of this low ash content in relation to the threshold values.

Part 2 – Biodegradation

Figure 14 visualizes the industrial composting procedure of the tested materials A and A-W. The biodegradation of the positive reference material (benchmark material that ensures the correctness of the test) was, as expected, at a high degree.
The biodegradation rates of both material samples were high during the first three weeks of testing. The biodegradation continued at the lower rate after the first three weeks, i.e. continued to evolve CO_2 at biodegradation rate. The biodegradation dynamics of the two tested materials are shown in figure 15.
Both tested material samples, A and A-W, biodegraded to approximately 50 % after 40 days (mean values).

After 180 days (6 months), the base material A was biodegraded to 85 %, thus not fulfilling the requirement of 90 % biodegradability within 6 months according to the EN 13432. From the diagram, it can be seen that the slopes of the curves continue to increase after 180 days, which is an indication of continued biodegradation. Possibly, the requirement of 90 % biodegradation could be met if the testing time had been extended.

material A – base material

material A-W – base mat./ welding finishing

composting test preparation

composting test preparations cont.

composting reactor: aerobic at 58°C
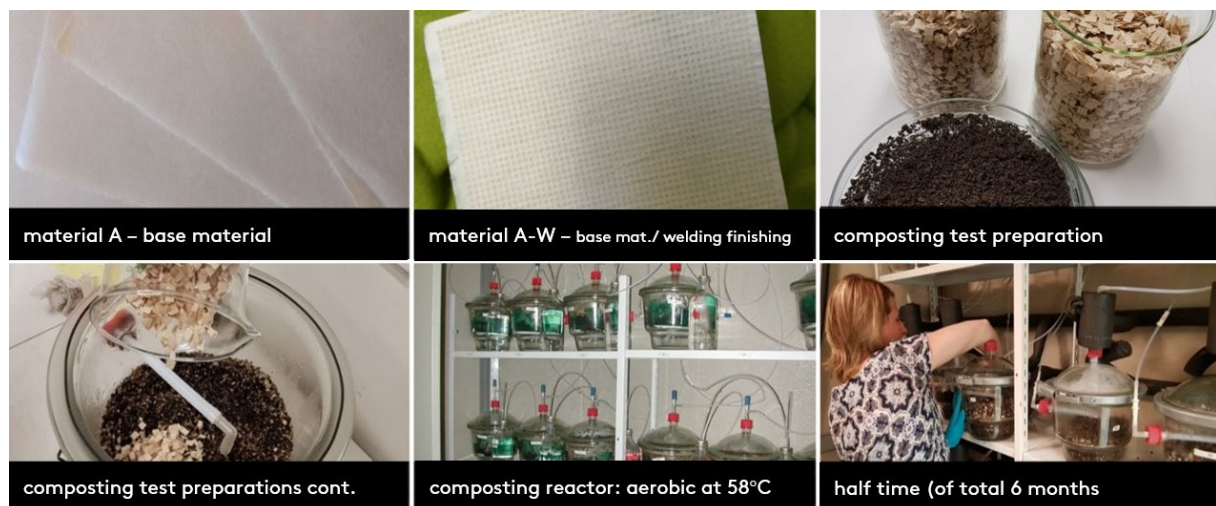
half time (of total 6 months

Figure 14: Composting process visualization.

The base material A-W with welding finishing was totally biodegraded and fulfills the requirement of 90 % biodegradability within 6 months according to EN 13432. A probable reason for the faster and higher biodegradability of the material A-W compared to the material A may be that the PLA fibers have been mechanically affected in the welding process, i.e. PLA fibers were degraded by the process heat. This led to shorter PLA polymer chains that are more

easily accessible for the microflora, being used by them as a substrate, thus leading to biodegradation at a higher rate. Hydrolyzation and microorganisms' metabolization occurred.
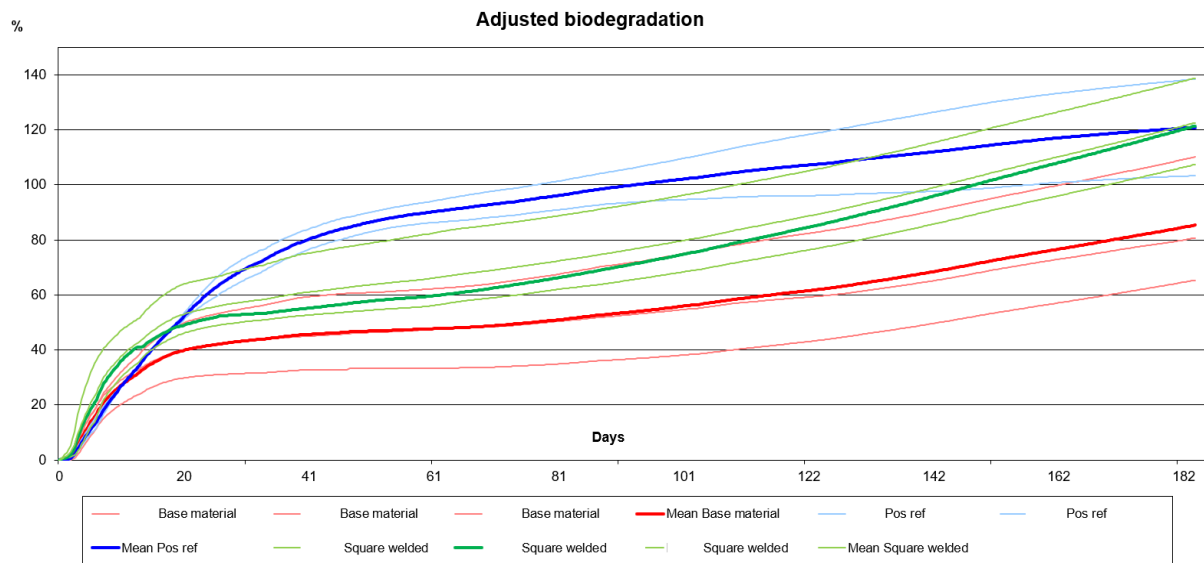
Adjusted biodegradation

figure 15: Biological degradation of material samples A and A-W during six-month period (180 days).

It should be noted that the materials A and A-W were produced at a smaller pilot so the reason for the large spread in the industrial composting results for each material may also be due to the fact that the materials were inhomogeneous in quality.

It should be also noticed that the biodegradation of the positive reference material and the material A-W was higher than 100%, which often can be seen in this kind of tests. An explanation for this known phenomenon may be the "priming effect" of organic matter which was reported by S. Fontaine et al[6]. A summary of the priming effect is as follows:

Addition of substrates with readily available carbon provides a rapid increase in various types of microbes which secrete enzymes. This higher content of microbes and enzymes enables the inaccessible carbon complexes in the soil to degrade faster. The total biodegradation can then be higher than 100%.

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[6] S. Fontaine, A. Mariotti, L. Abbadie, The priming effect of organic matter: a question of microbial competition?, 2003

4. conclusions

The idea behind performing the material recycling and industrial composting tests was to study how to maximize materials' cyclability potential already in the early material research & development (R&D) stage. One dimension was to test the 'extreme' proportions of PLA, 5% and 57% in the material, and another dimension was to compare the performance of the respective base materials contra the base materials treated with welding finishing.

The research was explorative in nature. It was envisioned that the route A-materials (high PLA content) could be industrially composted and that the route B-materials (low PLA content) could be materially recycled as a paper or paper board[1].

The results of the material recycling test through re-slushability showed that both base materials, A and B, were re-slushable (99,9%), meaning that such material can be recycled as paper packaging, i.e. disintegrated back into fibers at a recovered paper mill. It was easy to disintegrate textile-like paper back into its fiber components since the fibers were not chemically bonded to each other.

Material recyclability of the treated materials, however, was strongly influenced by the PLA content in the material. While the material B-W, containing low amount of PLA fibers and treated with welding finishing was re-slushable (99,64%), the material A-W, containing high amount of PLA fibers and treated with welding finishing showed worse result, being 55,3% re-slushable. This is due to the fact that PLA fibers have melted in the welding finishing process, which in turn made the textile-like paper fibers less available for disintegration and subsequent screening through the mesh with 0.15 mm slits. Further analysis of the reject fraction revealed 61% PLA content, while the rest were paper fibers attached to the smelted PLA.

According to EN 13432 it is required that 90% of the material is broken down within 6 months in order the material to be classified as biodegradable. The base material A with higher PLA content and welded finishing successfully biodegraded during the six-month time period.

The untreated base material biodegraded by slower pace and to extent of 85%, within six months. Therefore, it cannot be classified as biodegradable according to EN 13432. After analyzing the biodegradation dynamics of the material, it can be concluded that this material would need more time than six months to biodegrade 90%.

As a whole, the factor that hindered material recyclability within the project scope was a combination of a higher content of PLA and welding finishing, at the same time this factor contributed to the success of the industrial composting test. The test results are summarized in table 8 below:

table 8 results from material recyclability

Material samples	Material recycling through re-slushability (%)	Biodegradation in industrial composting test (% CO_2 generation during 6 months, mean values)
A	99,9	85
A-W	55,3	>100
B	99,9	Not tested
B-W	99,64	Not tested

The material recycling and industrial composting tests demonstrated that it is important to take a systemic approach in the material design. By systemic approach it is meant:

- Relevant-for-the-purpose choice of material components,
- Material design, including subsequent treatment processes in order to achieve specific material properties
- Designed for a fast user cycle
- Efficient cyclability of materials depending on the combinations of above and material application.

recommendations

Current study gave first insights into cyclability of the textile-like materials. It is recommended to continue the research and development according to the following:

- Perform full-scale industrial compostability study of textile-like materials with different PLA content, different finishing techniques (e.g. other welding patterns, crimping etc) and color treatments
- Study possibilities for separation of PLA fibers from paper fibers after re-slushability test and their potential uses
- Consider whether PLA-component in the material be replaced with another component
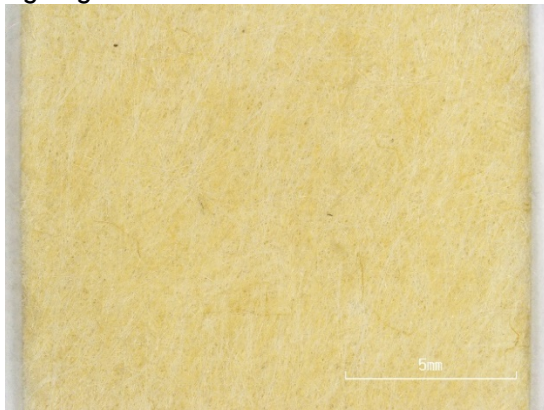
appendix

The screening accelerated ageing study included visual analysis of the material samples A-1 – A-6 and analysis of their mechanical properties.
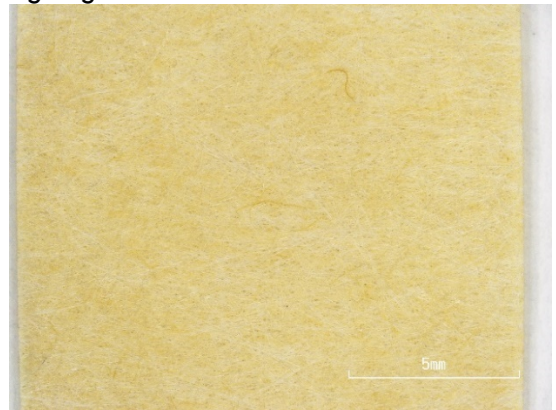

Ageing: 0h – machine direction
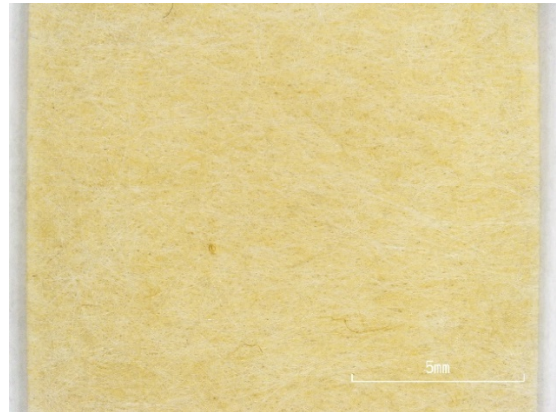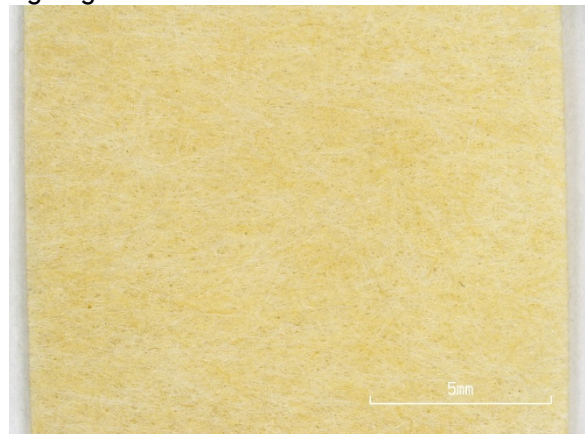

Ageing: 0h – cross direction

Ageing: 500h – machine direction

Ageing: 500h – cross direction

Ageing: 1000h – machine direction

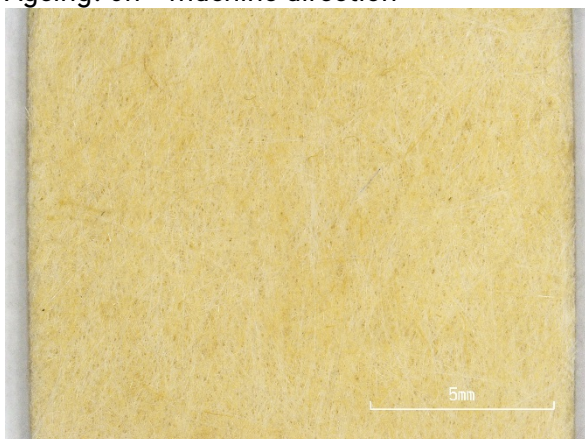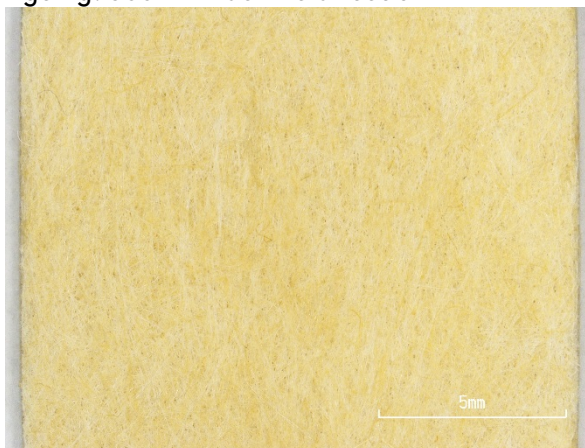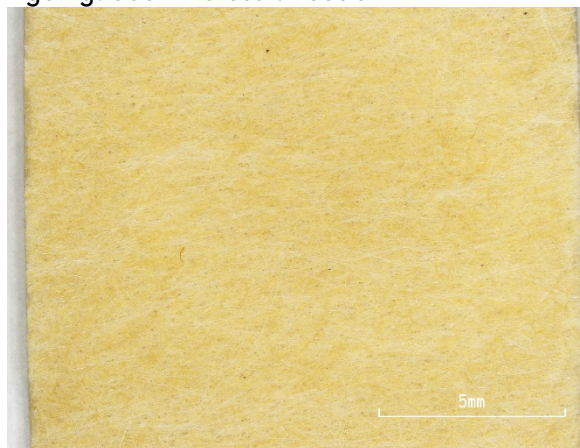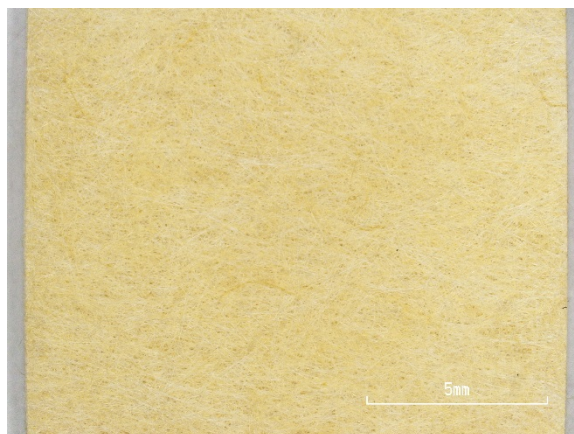
Ageing: 1000h – cross direction

Figure 16 Visual analysis of the aged material sample A-2 with grammage of **76,6 g/m²**. No visual differences observed.

Ageing: 0h – machine direction

Ageing: 0h – cross direction

Ageing: 500h – machine direction

Ageing: 500h – cross direction

Ageing: 1000h – machine direction

Ageing: 1000h – cross direction

Figure 17 Visual analysis of the aged material sample A-3 with grammage of 162,1 **g/m²**. No visual differences observed.
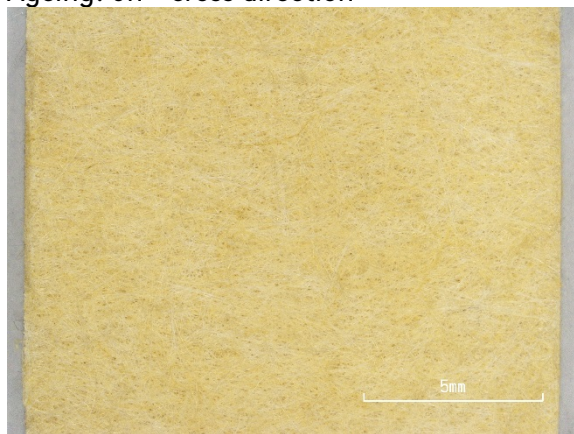
Ageing: 0h – machine direction

Ageing: 0h – cross direction

Ageing: 500h – machine direction

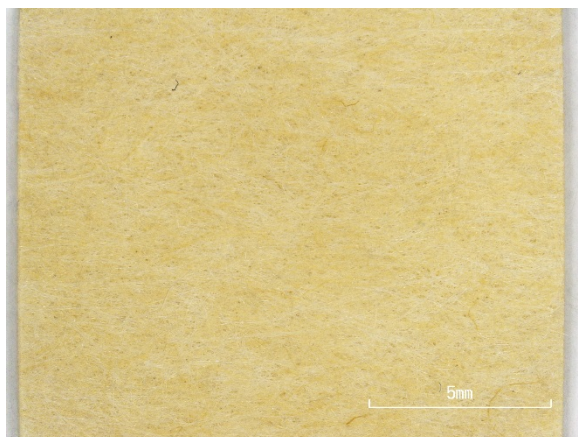Ageing: 500h – cross direction

Ageing: 1000h – machine direction

Ageing: 1000h – cross direction

Figure 18 Visual analysis of the aged material sample A-4 with grammage of 211,1 **g/m²**. No visual differences observed.
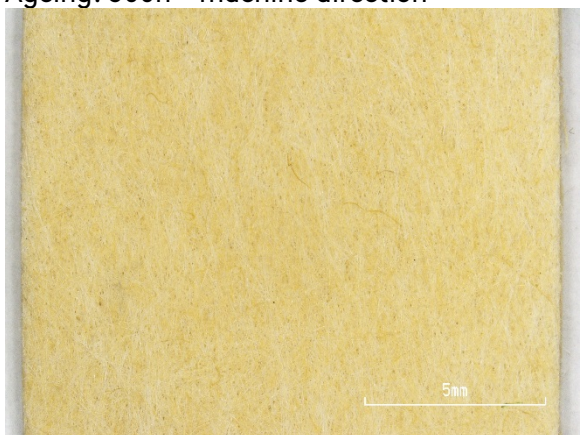
Ageing: 0h – machine direction

Ageing: 0h – cross direction

Ageing: 500h – machine direction

Ageing: 500h – cross direction

Ageing: 1000h – machine direction

Ageing: 1000h – cross direction

Figure 19 Visual analysis of the aged material sample A-6 with grammage of 229 **g/m²**. No visual differences observed.

Ageing: 0h – machine direction

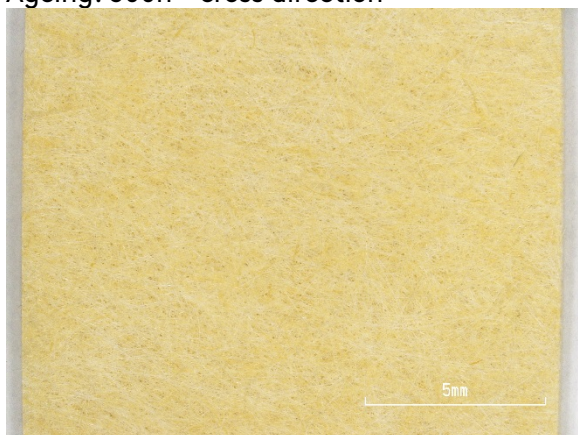Ageing: 0h – cross direction

Ageing: 500h – machine direction

Ageing: 500h – cross direction

Ageing: 1000h – machine direction

Ageing: 1000h – cross direction

Figure 20 Visual analysis of the aged material sample A-8 with grammage of 164,8 **g/m²**. No visual differences observed.

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Mistra Future Fashion is a research program that focuses on how to turn today's fashion industry and consumer habits toward sustainable fashion and behavior. Guided by the principles of the circular economy model, the program operates cross disciplinary and involves 60+ partners from the fashion ecosystem. Its unique system perspective combines new methods for design, production, use and recycling with relevant aspects such as new business models, policies, consumer science, life-cycle-assessments, system analysis, chemistry, engineering etc.

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